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IS : 6100 - 1984

Indian Standard

SPECIFICATION FOR SODIUM TRIPOLYPHOSPHATE, ANHYDROUS, TECHNICAL

(*First Revision*)

UDC 661.833.458

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 7 *February* 1985

AMENDMENT NO. 1 NOVEMBER 1986

TO

IS : 6100-1984 SPECIFICATION FOR SODIUM TRIPOLYPHOSPHATE, ANHYDROUS, TECHNICAL

(*First Revision*)

(*Page 15, clause B-8.4.2, line 3*) - Substitute the following for the existing matter:

'for 60 seconds between each addition, shake the flask for 15 minutes and then add the rest of the reagent in 1-ml portion, shaking for 60 seconds between each addition.'

(*Page 15, clause B-8.4.3, first sentence*) - Substitute the following for the existing matter:

'Reheat at 40°C and shake for 30 minutes.'

(CDC 3)

Indian Standard

SPECIFICATION FOR SODIUM TRIPOLYPHOSPHATE, ANHYDROUS, TECHNICAL

(*First Revision*)

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(*Continued on page* 26)

Indian Standard

SPECIFICATION FOR SODIUM TRIPOLYPHOSPHATE, ANHYDROUS, TECHNICAL

(*First Revision*)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 28 September 1984, after the draft finalized by the Inorganic Chemicals (Misc) Sectional Committee had been approved by the Chemical Division Council.

0.2 Sodium phosphate, especially the polyphosphate, are chemicals of wide application. The newest member of the family of important sodium phosphates is sodium tripolyphosphate. The tripolyphosphate exhibits most of the useful properties of the other phosphates and none of their undesirable ones. It has a high sequestering action and is a good deflocculant like pyrophosphate. At present, the largest use of sodium tripolyphosphate is as a builder of synthetic detergents. Most of the built detergents sold today contain sodium tripoly and/or pyrophosphate. Among other important uses of sodium tripolyphosphate are an ingredient of the formulation of boiler feed water treatment, purification of china clay, conditioning of oil drilling mud, antipith agent in paper making, textile processing, dispersant in cements, etc.

0.3 Sodium tripolyphosphate is manufactured by neutralizing the phosphoric acid produced either by wet process or by thermal process. It is made from a mixture of one mole of monosodium orthophosphate to two moles of disodium orthophosphate, which is intimately mixed to get a good yield of the desired product. If the mixing is inadequate, combinations of pyro-, tripoly- and metaphosphates are formed.

0.4 Currently the material in this country is being produced from phosphoric acid made by the wet process from imported rock phosphate. Keeping this in view the requirements have been laid down.

0.5 This standard contains clause **2.4** which calls for agreement between the purchaser and the supplier.

0.6 The standard was originally published in 1971 and is revised in the light of the progress made by the industry. In this revised standard, changes have been made in the requirement for sieve analysis and bulk density. The procedure for the gravimetric method for the determination of tripolyphosphate content has been amended while the volumetric method has been deleted in this revision.

0.7 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for sodium tripolyphosphate, anhydrous, technical, for use as a detergent builder, and as a deflocculant.

2. REQUIREMENTS

2.1 Description — The material shall consist essentially of sodium tripolyphosphate ($Na_5P_3O_{10}$). It shall be a white uniform free-flowing powder, free from visible impurities.

2.2 Sieve Analysis — When tested by the method prescribed in Appendix A, the material shall conform to the following size grading :

Sieve Size	*Percent by Mass*
Retained on 500-micron IS Sieve	0.2, *Max*
Passing through 500-micron IS Sieve but retained on 150-micron IS Sieve	20, *Max*
Passing through 150-micron IS Sieve and retained on 75-micron IS Sieve	20, *Max*
Passing through 75-micron IS Sieve	60, *Min*

2.3 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in Appendix B. Reference to the relevant clauses of Appendix B is given in col 4 of the table.

2.4 Temperature Rise — The temperature rise, when determined by the method prescribed in Appendix C, shall be as agreed to between the purchaser and the supplier.

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*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR SODIUM TRIPOLYPHOSPHATE, ANHYDROUS, TECHNICAL

(*Clause* 2.3)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. OF APPENDIX B)
(1)	(2)	(3)	(4)
i)	Moisture, percent by mass, *Max*	1.0	B-2
ii)	Matter insoluble in water, percent by mass, *Max*	0.1	B-3
iii)	Phosphorus (as P_2O_5), percent by mass, *Min*	56.0	B-4
iv)	*p*H value	9.5 to 10.1	B-5
v)	Iron (as Fe_2O_3), percent by mass, *Max*	0.025	B-6
vi)	Copper (as Cu), parts per million, *Max*	3	B-7
vii)	Tripolyposphate content (as $Na_5P_3O_{10}$), percent by mass, *Min*	90.0	B-8
viii)	Chlorides (as NaCl), percent by mass, *Max*	1.25	B-9
ix)	Fluorides (as NaF), parts per million, *Max*	600	B-10
x)	Bulk density, g/ml	0.8 to 1.2	B-11

NOTE — The analysis is done on as such basis and the results are expressed on dry basis.

3. PACKING AND MARKING

3.1 Packing — Unless agreed otherwise between the purchaser and the supplier, the material shall be packed in clean and sound paper bags.

3.2 Marking — Each bag shall be suitably marked with the following information :

a) Name and grade (anhydrous, technical) of the material;
b) Name of the manufacturer and/or his trade-mark, if any;
c) Batch number to enable the lot of manufacturer to be traced back from records;
d) Net mass; and
e) Year of manufacture.

3.2.1 The bags may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative samples of the material, the number of tests to be performed and the criteria for conformity of the material to the requirements of the specification shall be as prescribed in Appendix D.

APPENDIX A

(*Clause* 2.2)

SIEVE ANALYSIS

A-1. PROCEDURE

A-1.1 Clean and thoroughly dry the specified sieves. Nest the sieves in order with 500-micron IS Sieve on top. Weigh accurately 100 g of the well-mixed material and transfer it to the top sieve. Shake the sieves simultaneously and occasionally tap the bottom sieve on a rubber cushion. When the portion of the material passing through the bottom sieve appears to be negligible, remove the top sieve and shake the sieve separately over a clean glazed paper to ensure that no more of the material passes through. Transfer from the paper any material that has passed through 500-micron IS Sieve to 150-micron IS Sieve using a camel hair brush. Repeat the procedure with 150-micron IS Sieve and 75-micron IS Sieve. Transfer the residue on the various sieves to convenient tared containers with the aid of a camel hair brush and weigh each separately.

A-1.2 Calculation — Calculate the masses on the basis of percent by mass of the material.

APPENDIX B

(*Clause* 2.3)

METHODS OF TEST OF SODIUM TRIPOLYPHOSPHATE, ANHYDROUS, TECHNICAL

B-1. QUALITY OF REAGENTS

B-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

B-2. DETERMINATION OF MOISTURE

B-2.1 Procedure — Weigh accurately about 5 g of the material in a clean wide-mouth glass weighing bottle, previously dried and weighed. Place the weighing bottle in an oven maintained at 150 ± 2°C for two hours. Cool in a desiccator and weigh with the lid on. Heat again for 30 minutes, cool and weigh. Repeat this process until the loss in mass between the successive weighings is less than one milligram. Record the lowest mass obtained. Reserve the dried material for subsequent tests.

B-2.2 Calculation

$$\text{Moisture, percent by mass} = \frac{100\,(M_1 - M_2)}{M_1 - M}$$

where

M_1 = mass in g of the weighing bottle with the sample before drying,

M_2 = mass in g of the weighing bottle with the simple after drying, and

M = mass in g of the empty weighing bottle.

B-3. DETERMINATION OF MATTER INSOLUBLE IN WATER

B-3.1 Procedure — Weigh accurately about 10 g of the material and dissolve it in 200 ml of water and boil for about 10 minutes. Filter the hot solution through previously dried and weighed sintered glass crucible G No. 3 or asbestos padded Gooch crucible. Wash the crucible with hot water till the filtrate is free from alkali. Place the crucible in an oven maintained at 110 ± 2°C for two hours. Cool the crucible in a desiccator and weigh.

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*Specification for water for general laboratory use (*second revision*).

B-3.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = \frac{100\,(M_2 - M_1)}{M}$$

where

M_2 = mass in g of the crucible after filtration,
M_1 = mass in g of the crucible before filtration, and
M = mass in g of the material taken for the test.

B-4. DETERMINATION OF PHOSPHORUS

B-4.0 Outline of Method — Total phosphorus is determined volumetrically by freshly hydrolyzing the material with dilute hydrochloric acid and titrating with sodium hydroxide solution.

B-4.1 Apparatus

B-4.1.1 *Beaker* — 250 ml capacity, marked at the 100-ml level.

B-4.1.2 *pH Meter*

B-4.2 Reagents

B-4.2.1 *Dilute Hydrochloric Acid* — 5 N.

B-4.2.2 *Buffer Solution* — *p*H 10.

B-4.2.3 *Mixed Indicator Solution* — Mix equal volumes of bromocresol green (0.1 percent in water or 20 percent alcohol), phenolphthalein (0.5 percent in 60 percent alcohol), and absolute alcohol.

B-4.2.4 *Sodium Hydroxide Solution* — 5 N approximately and 0.1 N freshly standardized.

B-4.3 Procedure — Weigh accurately about 3.5 g of the material into a beaker. Dissolve in water, transfer to a 500-ml volumetric flask, dilute to the mark and shake to mix. Pipette out 75 ml into a 250-ml beaker marked at the 100-ml level. Add 15 ml of dilute hydrochloric acid and dilute to 100 ml. Introduce a boiling rod, cover with a watch-glass, and boil gently for 30 minutes. Add 0.5 ml of the mixed indicator, and neutralize the solution with sodium hydroxide solution (5 N) to a blue-green colour. Add dilute hydrochloric acid (0.5 N) until the indicator changes to yellow (about 0.5 ml is usually needed).

B-4.3.1 Dilute to 100 ml, if necessary, and boil for 15 minutes to expel carbon dioxide. Cool to room temperature. Wash and remove cover glass and wash boiling rod and walls of the beaker. Dilute to 100 ml. Place the beaker on the *p*H-meter assembly. With thorough stirring

(preferably mechanical), add standard sodium hydroxide solution (0.1 N) until the *p*H reaches 4.1. Alternatively add the alkali in portions of about 0.2 ml, reading the burette after each addition, and interpolate to find the burette reading corresponding to *p*H 4.1.

B-4.3.2 Titrate to *p*H 8.8 with standard sodium hydroxide solution (0.1 N) interpolating as above, if desired. The final volume shall be about 150 ml; hence, excessive rinsing of the burette tip, electrodes, etc, should be avoided.

B-4.4 Calculation

$$\text{Total phosphorus (as } P_2O_5 \text{)} = \frac{T \times f}{10\,000} \times 70{\cdot}98 \times \frac{500}{75} \times \frac{100}{M}$$

$$\frac{T \times f \times 4{\cdot}732}{M}$$

where

T = difference in volume in sodium hydroxide solution required in **B-4.3.1** and **B-4.3.2,**

f = factor of N/10 sodium hydroxide solution (*see* **B-4.5**), and

M = mass in g of the material taken for the test.

B-4.5 Standardization of N/10 Sodium Hydroxide Solution — Analyze a sample of pure anhydrous sodium pyrophosphate ($Na_4P_2O_7$) by proceeding exactly as above except for accurately weighing 3.8 g of sample instead of 3.5 g.

$$\text{Total phosphorus (as } P_2O_5 \text{)} = \frac{T \times f \times 4{\cdot}732}{M} = 53{\cdot}38$$

$$\frac{53{\cdot}38 \times M}{4{\cdot}732 \times T}$$

where

T and f = same as in **B-4.4,** and

M = mass in g of pure sodium pyrophosphate taken.

NOTE — If pure anhydrous sodium pyrophosphate is not available, pure potassium dihydrogen orthophosphate crystals may be used for standardization of N/10 sodium hydroxide solution, then f would be

$$\frac{52{\cdot}17 \times M}{4{\cdot}732 \times T}$$

B-5. DETERMINATION OF *p*H

B-5.1 Apparatus

B-5.1.1 *p*H *Meter* — A suitable *p*H meter fitted with glass and calomel electrodes.

B-5.2 Procedure — Weigh 1.0 g of the material into a 150-ml beaker, add 100 ml of freshly boiled and cooled water and stir to dissolve. Measure the *p*H of the solution after 10 minutes with the help of a *p*H meter.

B-6. DETERMINATION OF IRON

B-6.0 Outline of Method — Iron is determined colorometrically by measuring the transmittancy of reddish purple colour produced by ferrous and ferric ion with thioglycollic acid at *p*H 10.

B-6.1 Apparatus

B-6.1.1 *Photoelectric Absorptiometer*

B-6.2 Reagents

B-6.2.1 *Dilute Hydrochloric Acid* — 5 N.

B-6.2.2 *Sodium Citrate Solution 1 M* — 29.4 g per 100 ml. Alternatively, ammonium citrate solution (2 M), 43.0 g per 100 ml, may be used.

B-6.2.3 *Ammonium Hydroxide* — 10 N.

B-6.2.4 *Thioglycollate Reagent* — Add 20 ml of ammonium hydroxide to 30 ml of water, then add a mixture of 10 ml of thioglycollic acid and 40 ml of water.

B-6.2.5 *Standard Iron Solution* — Dissolve 0.4911 g of ferrous ammonium sulphate [$FeSO_4 (NH_4)_2 SO_4.6H_2O$] in water, add 10 ml of dilute sulphuric acid (10 percent *v/v*). Dilute to exactly 100 ml in a volumetric flask. Take 25 ml of this solution into a 250-ml volumetric flask and dilute to the mark with water. One millilitre of this solution is equivalent to 0.1 mg of iron (as Fe_2O_3).

B-6.3 Procedure — Weigh accurately 1 g of the material into a 150-ml beaker. Add 20 ml of water and 5 ml of dilute hydrochloric acid. Introduce a boiling rod, cover with a watch-glass and boil gently for 15 to 20 minutes. Cool and transfer to a 100-mI volumetric flask and dilute to about 70 ml. Place 5 ml of dilute hydrochloric acid and 65 ml of water in a second flask as blank.

B-6.3.1 To each flask add 4 ml of sodium citrate solution (or 2 ml of ammonium citrate solution), 5 ml of thioglycollate reagent and 15 ml of ammonium hydroxide, swirling after each addition. Dilute to the mark and mix well.

B-6.3.2 Balance the absorptiometer at blank using green filter No. 4 and 4-cm cells. (Use 1-cm cells if the colour is too strong). Find the reading with the sample solution. Read off milligram iron from the standard calibration graph.

B-6.3.3 *Standard Calibration Graph* — Pipette known volumes of standard iron solution into each of 100-ml volumetric flasks containing 5 ml of dilute hydrochloric acid. Proceed as in **B-6.3.1.** Suitable aliquots are 0, 1.0, 1.5, 2.0, 2.5 and 3.0 ml. Take the readings. Plot graph with milligram iron as ordinates and absorptiometer readings as abscissae. Draw a line through points and extend through the origin. Label the graph with particulars of filters and cells used.

B-6.4 Calculation

$$\text{Iron (as } Fe_2O_3 \text{), percent by mass} = \frac{M_1}{M}$$

where

M_1 = mass in mg of iron (Fe_2O_3) read off from the graph, and

M = mass in g of the material taken for the test.

B-7. DETERMINATION OF COPPER

B-7.0 Outline of Method — Copper is determined colorimetrically by measurement of the colour produced by the material with zinc dibenzyldithiocarbomate. The value is read off from a standard calibration graph.

B-7.1 Apparatus

B-7.1.1 *Separating Funnels* — 100 ml capacity. Rinse the funnel with dilute hydrochloric acid and with water before use. Allow to drain, then dry the stems with pure absorbent cotton wool and place a small plug of cotton wool in each stem.

B-7.1.2 *Photoelectric Absorptiometer* — with 1-cm cells.

B-7.2 Reagents

B-7.2.1 *Ammonium Hydroxide* — 10 N approximately.

B-7.2.2 *Dilute Hydrochloric Acid* — 5 N.

B-7.2.3 *Zinc Dibenzyldithiocarbomate Solution* — 0.04 percent solution in carbon tetrachloride.

B-7.2.4 *Standard Copper Solution* — Weigh 0.491 g of copper sulphate pentahydrate ($CuSO_4, 5H_2O$) in a weighing bottle. Wash into a 250-ml volumetric flask. Add 3 ml of 18 N sulphuric acid, dilute to about 100 ml, and swirl until the salt has completely dissolved. Dilute to the mark and mix well. This is the stock solution, one millilitre of which contains 0.5 mg of copper (Cu). Take 3 ml of 18 N sulphuric add and about 200 ml of water in a 1 000-ml volumetric flask. Carefully pipette 5 ml of the stock copper solution into the flask. Dilute to the mark and mix well. One millilitre of this solution contains 2.5 µg of copper (Cu).

B-7.3 Procedure

B-7.3.1 Weigh accurately 5 g of the material into a 250-ml beaker. Take a, smaller mass if the copper is expected to exceed 3 parts per million. Add 30 ml of dilute hydrochloric acid and boil gently for 15 minutes. Place 30 ml of dilute hydrochloric acid in a second beaker as blank and continue with both. Allow to cool, then add ammonium hydroxide until the solution is alkaline to litmus paper. Add dilute hydrochloric acid dropwise until the solution is acidic, then add 3 ml in excess.

B-7.3.2 Transfer to a 100-ml separating funnel and, if necessary, dilute to 50 ml. Add 10 ml of zinc dibenzyldithiocarbomate solution and shake vigorously for at least 2 minutes. Allow the layers to separate, then unstopper the funnel, and run the lower layer through a plug of cotton wool into a 1-cm cell, using the first few drops to rinse out the cell. Adjust the photometer at zero at 440 μm (blue photocell, plain glass filter) with a cell of zinc dibenzyldithiocarbomate solution in the light path, then measure the sample and the blank. Read off parts per million copper from the standard calibration graph.

B-7.3.3 *Standard Calibration Graph* — Take five 100-ml separating funnels, and wash thoroughly with hydrochloric acid and then with water. In each funnel place 3 ml of dilute hydrochloric acid (5 N). Using pipettes or a micro burette, measure respectively 0, 2, 5, 10 and 15 ml of standard copper solution. Dilute to 50 ml and proceed as in **B-7.3.2.** Subtract a constant from each observed colorimeter reading so that the blank solution with no added copper corresponds to a reading of 0. Convert volumes of standard copper solution to micrograms of copper by multiplying by 2.5. Plot a graph with micrograms as ordinates and adjusted colorimeter readings as abscissae. Draw a line through the points and through the origin. Label the graph with particulars of cells and filters.

B-7.4 Calculation

$$\text{Copper (Cu), parts per million} = \frac{M_1}{M}$$

where

M_1 = mass in micrograms of copper read off from the graph, and

M = mass in g of the material taken for the test.

B-8. DETERMINATION OF TRIPOLYPHOSPHATE CONTENT ($Na_5P_3O_{10}$)

B-8.0 Outline of Method — The method is based on the reaction that sodium tripolyphosphate forms a complex with tris diaminoethane cobaltic chloride at *p*H 3.5 selectively in the presence of other phosphates.

B-8.1 Reagents

B-8.1.1 *Wash Solution* — Dissolve 20 g isopropanol in 100 ml of water.

B-8.1.2 *p-Nitrophenol Indicator Solution* — Dissolve 0.1 g of *p*-nitrophenol in 100 ml of water.

B-8.1.3 *Dilute Hydrochloric Acid* — 2 N approximately.

B-8.1.4 *Acetate Buffer Solution* - *p*H 3.6. Dissolve 52 to 53 ml of glacial acetic acid (*see* IS : 695-1972*) and 6.16 g of anhydrous sodium acetate in water and dilute to 500 ml.

B-8.1.5 *Trisencobalt Reagent* — Dissolve 10 g of tris diaminoethane cobaltic chloride (as prepared in **B-8.2**), after drying at 110°C, in water and dilute to 250 ml. Add 1 drop of toluene to prevent mould growth.

B-8.1.6 *Isopropyl Alcohol*

B-8.1.7 *Sodium Trypolyphosphate, Hexahydrate* — thrice crystallized as prepared in **B-8.1.3.**

B-8.2 Preparation of Tris Diaminoethane Cobaltic Chloride

B-8.2.1 *Reagents*

a) *Diaminoethane hydrate (ethylene diamine)*

b) *Concentrated hydrochloric acid* — *see* IS : 265-1976†.

c) *Cobalt chloride* ($CoCl_2.6H_2O$)

d) *Methylated spirit* — *see* IS : 324-1959‡.

B-8.2.1.1 *Procedure*

a) Weigh 261 g of diaminoethane hydrate into a 1 000-ml beaker. Partly neutralize with 85 ml of concentrated hydrochloric acid in 535 ml of water. Pour the mixture with good agitation into a solution of 250 g of cobalt chloride in 750 ml of water contained in a conical flask. Pass a vigorous stream of air into the solution through a gas distribution tube for 8 hours.

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*Specification for acetic acid (*second revision*).

†Specification for hydrochloric acid (*second revision*).

‡Specification for ordinary denatured spirit (*revised*).

b) Transfer the solution to a 2 000-ml beaker and evaporate on a steam-bath under a stream of air until crystals form on the surface of the solution. Cool the solution and add 150 ml of concentrated hydrochloric acid with good mechanical stirring. Warm until the crystals re-dissolve. Salt out the tris diaminoethane cobaltic chloride by slowly adding, over a period of 1 hour, 300 ml of methylated spirit to the mechanically stirred solution. Cool and filter through a Buchner funnel with suction. Discontinue suction and wash by slurrying with 150 ml of methylated spirit. Re-apply suction and filter. Repeat the washings three more times. Suck dry; spread out the crystals in an evaporating dish and allow the methylated spirit to evaporate.

c) Dissolve the crystals obtained in (b) above in 200 ml of boiling water in a 1 000-ml beaker on a hot-plate using mechanical stirring. If the crystals do not dissolve in this volume of water, add 20-ml portion of water, reheating to boiling after each addition, until complete solution is obtained. Remove from the source of heat and salt out with 300 ml of the methylated spirit as above. Filter and wash as before. If four washings do not give a colourless filtrate, continue washing until the filtrate is colourless.

d) Spread out the crystals in a thin layer in large evaporating dishes and allow to air-dry overnight. Then dry in an oven at 110°C overnight.

NOTE — These crystals are hygroscopic and should be freshly dried each time they are weighed to make a new solution. An yield of about 300 g of the dried crystals is obtainable.

B-8.3 Preparation of Pentasodium Trioplyphosphate, Hexahydrate ($Na_5P_3O_{10},6H_2O$)

B-8.3.1 *Procedure* — Weigh 150 g of commercial sodium tripolyphosphate, using material of low metaphosphate content. Dissolve in 1 200 ml of water, and filter to remove any suspended material. Add 300 ml of methylated spirit slowly, with mechanical stirring, over a period of 2 hours. Separate the crystals of hexahydrate by filtration using a Buchner funnel fitted with a glass disc of medium porosity. Wash the crystals successively with 100 ml each of 25 percent, 50 percent, 75 percent and 90 percent (*v/v*) solution of methylated spirit in water.

Dissolve the hexahydrate in 900 ml of water and salt out with 225 ml of methylated spirit using the same procedure as above, again, filtering and washing the crystals with the same volumes of dilute methylated spirit. Repeat the recrystallization using 675 ml of water and 170 ml of methylated spirit. Recrystallize once more using 500 ml of water and 150 ml of methylated spirit. After filtering and washing, air-dry the crystals of hexahydrate for several days.

NOTE — The yield is about 60 g of $Na_5P_3O_{10}$, $6H_2O$. Store the hexahydrate in a tightly stoppered bottle. The purification procedure should not be interrupted for any length of time unless the material is in a crystalline form, to avoid hydrolysis. Do not allow it to remain in solution overnight.

B-8.4 Procedure for Determination of Tripolyphosphate Content

B-8.4.1 Transfer 3.500 ± 0.003 g of the material to a beaker. Dissolve in water, transfer to a 500 ml graduated flask, dilute to the mark and mix well. Pipette 50 ml of this solution into a 250 ml conical flask. Add 4 drops of ρ-nitrophenol indicator, and add dilute hydrochloric acid dropwise until the solution is just acidic (that is, when the indicator becomes colourless). Add 10 ml of acetate buffer solution and 30 ml of isopropanol and then heat the solution to 40°C.

B-8.4.2 Transfer 10 ml of trisencobalt reagent into a clean 10-ml burette. Add four 1-ml portion of the reagent from the burette and shake for 60 seconds between each addition and then add the rest of the reagent in 1-ml portion, shaking for 60 seconds between each addition.

B-8.4.3 Keep at 40°C for 30 minutes. Filter through a medium porosity sintered glass crucible (which has previously been washed, dried and weighed).

B-8.4.4 Wash the precipitate with 2 percent isopropanol, wash solution to make it free from chloride (normally 6 to 8 times is required). Dry in an air oven at 98°C – 102°C for 1 to 2 hours, cool in a desiccator and weigh till constant mass.

B-8.5 Calculation

Sodium tripolyphosphate content (as $Na_5P_3O_{10}$), percent by mass $= 198.4 \times M$

where

M = mass in g of the precipitate obtained.

NOTE — When the analysis obtained indicates less than 85 percent of sodium tripolyphosphate, take a 25-ml aliquot and add 0.2264 g of pure tripolyphosphate hexahydrate crystals, washed with 25-ml of water. Multiply the result by two and deduct 100 percent. If appreciable amounts of phosphate glass are present and indeed in any case where precipitation does not occur when the first 4 ml of reagent are added, this modification should always be used.

B-9. DETERMINATION OF CHLORIDES'

B-9.0 Outline of Method — Chlorides are determined gravimetrically by precipitating with silver nitrate in nitric acid medium.

B-9.1 Reagents

B-9.1.1 *Concentrated Nitric Acid — see* IS : 264-1976*.

B-9.1.2 *Silver Nitrate Solution* – 0.1 N approx.

B-9.1.3 *Dilute Nitric Acid* — 0.02 N approx.

B-9.2 Procedure — Weigh about 5 g of the material and wash into a 400-ml beaker. Dilute to 150 ml with water, and add 3 ml of concentrated nitric acid. Warm and stir until the sample dissolves. Cover the vessel with thick brown paper, to minimize exposing the subsequent precipitate to light. Add from a pipette, with constant stirring, 50 ml of silver nitrate solution. Heat the solution to nearly boiling, while stirring constantly and maintain it at this temperature until the precipitate coagulates and the supernatant liquor is clear (2 to 3 minutes). Make certain that the precipitation is complete by adding a few drops of silver nitrate solution to the supernatant liquor. If no further precipitate appears, place the beaker in the dark and allow to stand for 1 hour. Filter the liquor through a tared sintered glass crucible G No. 4. Wash the precipitate three times by decantation with cold dilute nitric acid before transferring the precipitate to the crucible. Transfer the last particles with the aid of a rubber-tipped rod. Wash the precipitate at least ten times with small amounts of dilute nitric acid. Heat the crucible in an oven at 130°C to 150°C for 30 minutes. Cool in a desiccator and weigh. Repeat the operation till constant mass is obtained.

B-9.3 Calculation

$$\text{Chlorides (as NaCl), percent by mass} = \frac{M_1 \times 40{\cdot}8}{M}$$

where

M_1 = mass in g of the precipitate obtained, and

M = mass in g of the material taken for the test.

B-10. DETERMINATION OF FLUORINE BY DISTILLATION METHOD

B-10.0 Outline of the Method — Fluorine is separated as hydrofluosilicic acid by distillation with perchloric acid and determined by titration against standard thorium nitrate solution using sodium alizarine sulphonate as indicator.

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*Specification for nitric acid (*second revision*).

B-10.1 Apparatus

B-10.1.1 The distillation apparatus (*see* Fig. 1) consists of a 250 ml distillation flask *(A),* side arm of which is connected to a condenser (*B*), The neck of the flask is fitted with a two-holder rubber stopper flask to a steam generator *(E),* which is equipped with two discharges, one tube *(F),* to release steam and the other (*D)* to deliver steam into the distillation flask (*A*). The distillate is collected in a PVC beaker (500 ml).

B-10.1.2 *Nessler Cylinders* — 100 ml capacity with marks 100 ml and 50 ml

B-10.1.3 *Thermometer* — 0-150°C.

B-10.1.4 *Burette with Top* — 10 ml capacity, graduation interval 0.05 ml (*see* IS : 1997-1967*).

B-10.2 Reagents

B-10.2.1 *Perchloric Acid* — 70 percent.

B-10.2.2 *Sodium Alizarine Sulphonate Indicator* — Dissolve 0.02 g of sodium alizarine sulphonate in 100 ml of water and store in bottle.

B-10.2.3 *Sodium Hydroxide Solution* — Dissolve 10 g of sodium hydroxide pallets in water, cool and dilute to one litre. Mix well.

B-10.2.4 *Dilute Hydrochloric Acid* — Dilute 19.6 ml of concentrated hydrochloric acid to 1 000 ml with water. Mix well.

B-10.2.5 *Standard Thorium Nitrate Solution* — 0.02 N. Dissolve 2.940 9 g of thorium nitrate, $Th(NO_3)4.6H_2O$ in water and make up to one litre. Mix well.

B-10.2.6 *Standard Sodium Fluoride Solution (1 ml = 0.1 mg NaF)* — Dissolve 1.0 g of sodium fluoride (AR) previously dried at 120°C to constant mass in one litre of water, in a volumetric flask, and further dilute 10 ml of this solution to 100 ml containing 20 ml of 1 N NaOH. This is the standard solution (1 ml = 0.1 mg NaF) which can be used for titration. Keep the solution in polyethylene bottle.

B-10.3 Procedure

B-10.3.1 Transfer 10.0 g of an accurately weighed sample to a 250 ml distillation flask, containing a few glass beads. Add 10 ml of perchloric acid.

---

*Specification for burettes (*first revision*).

B-10.3.2 Place a 500 ml PVC beaker under the condenser. Connect the apparatus as shown in Fig. 1, distillation flask (*A*) to the condenser and distil the solution until the boiling point reaches 140°C. While this is being done, heat the water in the steam generator (*E*) to boiling but do not allow the steam to get in the flask (*A*). When the temperature reaches 140°C admit steam into the flask (*A*). Maintain the temperature 140°C ± 2°C by adjusting the steam flow and heating of the flask (*A*). Distil at such a rate that no fumes or mist escape from the PVC beaker. Continue distillation until nearly 300 ml are collected in a beaker.

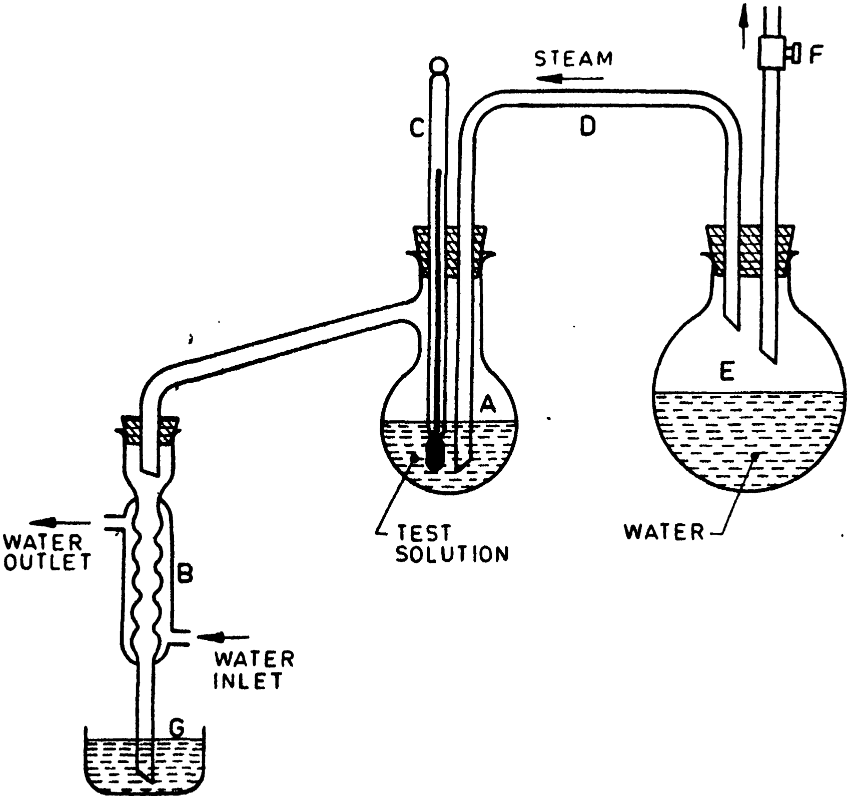


FIG. 1 APPARATUS FOR STEAM DISTILLATION FOR FLUORIDE DETERMINATION

B-10.3.3 Transfer to a 500 ml volumetric flask. Make the volume upto the mark and mix well. Pipette out 50 ml aliquot into a 150 ml beaker.

Take 10 ml of standard sodium fluoride (1 ml = 0.1 mg) solution in another 150 ml. Add 1 ml of sodium alizarine sulphonate indicator in each beaker. Adjust the *p*H of both the solution on *p*H meter to 3.1 using sodium hydroxide or dilute hydrochloric acid as the case may be. Transfer both the solutions into two different Nessler cylinders. Add thorium nitrate (0.020 N) solution from burette in standard fluoride solution until light pink colour appears without the formation of precipitate (suitable for comparison). Note the reading. Add thorium nitrate in sample solution till it matches in colour developed with standard fluoride solution. Again note the reading.

B-10.4 Calculation

$$\text{Sodium fluoride, parts per million} = \frac{T}{1\,000} \times F \times \frac{500}{50} \times \frac{1\,000\,000}{M}$$

$$= \frac{V \times F \times 10\,000}{M}$$

where

V = volume in ml of thorium nitrate required for sample solution,

$F = \dfrac{\text{mg of NaF taken for comparison}}{\text{ml of thorium nitrate added to it}}$, and

M = mass in g of sample taken for distillation.

B-11. BULK DENSITY

B-11.1 Apparatus

B-11.1.1 The apparatus is shown in Fig. 2. The base of the measuring cylinder *A* shall be ground flat and the empty measuring cylinder *A* together with the rubber bung shall weigh 250 ± 5 g. It shall be accurately calibrated to 250 ml with an error of less than one millilitre. The distance between zero and 250-ml graduation on the measuring cylinder *A* shall be not less than 220 mm and not more than 240 mm. The distance between the flat-ground part of the base of the measuring cylinder *A* and the rubber base pad *B*, when the measuring cylinder *A* is raised to the full height shall be 25 ± 2 mm.

B-11.1.2 *Balance* — Pans of the balance shall be at least 10 cm in diameter and the balance shall be sensitive to less than 0.1 g.

B-11.1.3 *Rubber Base Pad* — *B* shall have a shore hardness of 35 to 50.

B-11.2 Procedure — Weigh 100 g of the material in a paper and form it into **a** chute. Introduce the powder into the cylinder, which should be held at 45° to the vertical. Slip the powder gently and smoothly into the measuring cylinder without knocking or squeezing. Assemble the apparatus. With the thumb and four fingers of the hand, gently grasp the upper part of the cylinder and within one second lift it as far as 25 mm. Do not pick by knocking it against the upper stop. At the start of the next second, release the cylinder smoothly. Continue lifting and dropping until 50 complete drops have been given. Once in every 2 seconds a gentle twist of 10° should be given to the cylinder. Read off the volume.

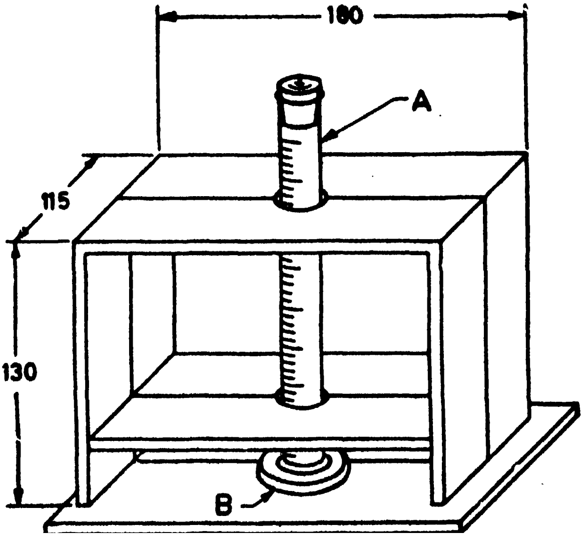
B-11.3 Calculation

$$\text{Bulk density (g/ml)} = \frac{M}{V}$$

where

M = mass in g of the material taken for the test, and

V = final volume in ml of the material.



All dimensions in millimetres.

FIG. 2 APPARATUS FOR DETERMINATION OF BULK DENSITY

APPENDIX C

(*Clause* 2.4)

DETERMINATION OF TEMPERATURE RISE

C-1. GENERAL

C-1.1 There are two types of sodium tripolyphosphate, namely, Phase I and Phase II, both of which are found in commercial samples. The ratio of Phase I to Phase II is determined by an empirical test based on the fact that Phase I hydrates more rapidly than Phase II. This test, called the temperature rise test has been calibrated by X-ray analysis.

C-2. APPARATUS

C-2.1 Thermos Flask — wide-mouth vacuum flask, capacity about 1 200 ml; inner dia about 8 cm; height about 22 cm.

C-2.2 Beaker — tall form 200-ml; height 10 cm; dia 6.5 cm.

C-2.3 Stirrer — a stirrer having two 3.5 cm diameter rings attached 2.5 cm apart to a handle and made from 0.3 cm stainless steel rod.

C-2.4 Thermometers — two thermometers graduated from 0 to 50°C, least count 0.1 °C and total length 550 mm (*see* schedule mark 22 of IS : 4825-1968*).

C-2.5 Stop-Watch

C-2.6 Measuring Cylinder — 25 ml capacity.

C-3. REAGENTS

G-3.1 Glycerine — Purity 96.5 ± 0.5 percent; specific gravity at 20°/20°C, 1.254 6 ± 0.001 5.

C-4. PROCEDURE

C-4.1 Weigh 50.0 ± 0.1 g of glycerine into the dry beaker at room temperature and put 50.0 ± 0.1 g powdered sample at room temperature on top of the glycerine. Suspend the beaker in the neck of vacuum flask and insert the dry stirrer to the bottom of the beaker (*see* Fig. 3).

C-4.2 Stir with vertical strokes for 30 seconds at the slow speed (*see* Note under **C-4.4**) (to prevent dusting and loss of sample and to mix powder and glycerine properly). Then increase the stirring rate quickly to fast speed (*see* Note under **C-4.4**) and stir for 2½ minutes.

---

*Specification for laboratory and reference thermometer.

C-4.3 Stop stirring. Clamp the dry thermometer in a central position with the tip 10 mm above the bottom of the beaker (lower it until it touches the bottom and lift 10 mm). Read and record the highest temperature of the paste that is constant for 30 seconds (T_1).

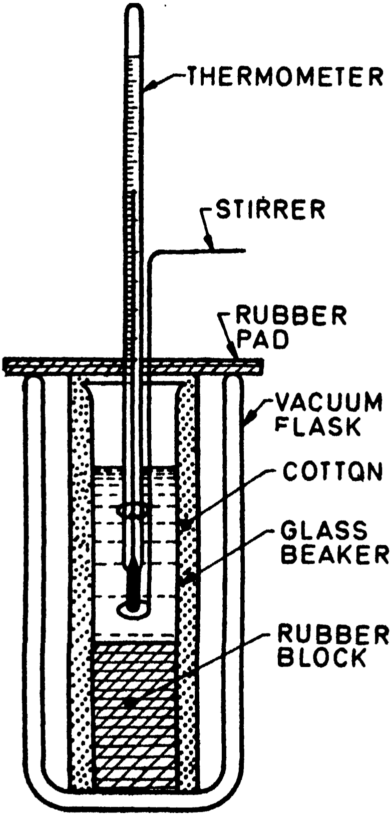


FIG. 3 APPARATUS FOR DETERMINATION OF TEMPERATURE RISE

C-4.4 Quickly add 25 ml of water at temperature (T_1) within ± 0.1°C and then stir at slow speed for 5 seconds and then at fast speed for 1 minute. Push the stirrer down to the bottom of the beaker. Observe the temperature without stirring until it has reached a maximum and decreased again at least 0.1°C (T_2).

NOTE — Slow speed : about 90 complete cycles/minute.
Fast speed : about 240 complete cycles/minute.

A complete cycle always means lifting up the bottom ring of the stirrer from the bottom of the beaker just to the surface of the mixture and pushing it back down until it touches the bottom again. Scrapping of the sides is also desirable.

C-5. CALCULATIONS

C-5.1 Temperature rise (deg C) $= T_2 — T_1$

where

T_2 = maximum temperature deg C, and
T_1 = paste temperature deg C.

C-5.2 Phase I, percent by mass = (Temperature rise deg C — 6)*.

APPENDIX D

(*Clause* 4.1)

SAMPLING OF SODIUM TRIPOLYPHOSPHATE, ANHYDROUS, TECHNICAL

D-1. GENERAL REQUIREMENTS OF SAMPLING

D-1.0 In drawing, preparing, storing and handling the samples, the following precautions shall be observed.

D-1.1 Samples shall not be taken at a place exposed to the adverse effects of weather.

D-1.2 The sampling instruments and sample containers shall be clean and dry,

D-1.3 Before drawing the samples, the contents of the selected containers shall be thoroughly mixed.

D-1.4 After filling, the sample containers shall be sealed and marked with relevant particulars.

D-2. SCALE OF SAMPLING

D-2.1 All the containers in a consignment of the material drawn from a single batch of manufacture shall constitute a lot. Each lot shall be tested separately for all the requirements of the specification.

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*This is an empirical relation.

D-2.2 The number of containers to be sampled at random from a lot depends on the size of the lot and shall be as given in Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

LOT SIZE (1)	No. OF CONTAINERS TO BE SELECTED IN THE SAMPLE (2)
Up to 50	3
51 „ 100	4
101 „ 150	5
151 and above	7

D-3. PREPARATION OF TEST SAMPLES

D-3.1 Draw with an appropriate sampling implement about 100 g of the material from different parts of each of the selected containers. These quantities are called individual samples representing the selected containers. These individual samples shall be kept in different sample containers which shall be marked with full details of sampling.

D-3.2 Equal quantities from each of the individual samples shall be taken and mixed together to form a composite sample weighing approximately 200 g.

D-4. NUMBER OF TESTS

D-4.1 Tests for the determination of phosphorus content and matter insoluble in water shall be conducted on each of the individual samples.

D-4.2 Tests for the remaining characteristics shall be conducted on the the composite sample.

D-5. CRITERIA FOR CONFORMITY

D-5.1 For Individual Samples — From the test results Mean (X) and Range (R) shall be calculated.

$$\text{Mean}\,(X) = \frac{\text{Sum of the test results}}{\text{Number of tests}}$$

Range (R) = Difference between the maximum and minimum value of the test results.

D-5.1.1 For phosphorus content the lot is deemed to conform to the specification if $X - 0.6\ R$ is not less than the minimum value given in Table 1 and for matter insoluble in water the lot is deemed to conform to the specification if $X + 0.6\ R$ is not greater than the maximum value given in Table 1.

D-5.2 For the remaining characteristics, all the test results shall satisfy the corresponding requirements given in col 3 of Table 1.

(*Continued from page* 2)

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